REAL-TIME SENSING OF METALLURGICAL TRANSFORMATIONS BY LASER-ULTRASOUND

S. E. Kruger, A. Moreau, C. Bescond and J.-P. Monchalin
Industrial Materials Institute - NRC, Boucherville, Quebec, Canada

Abstract: The ability of laser-ultrasound to monitor in real-time metallurgical transformations is reviewed in this paper. Although the technique is appropriate for online industrial materials characterization, this paper addresses laboratory characterization of metallurgical transformations. Examples are presented on the real-time sensing of grain size and grain growth, phase transformation and recrystallization of aluminum and steel. Results show good sensitivity of the laser-ultrasonic technique to microstructural parameters. The comparison with traditional in-situ (eg. dilatometry) or ex-situ (eg. metallography) characterization techniques is discussed. Experimental configurations for a cost-effective laser ultrasonic system coupled to a laboratory furnace and more specifically to a Gleeble thermo-mechanical simulator are discussed.

Introduction: The use of laser-ultrasonics to the monitoring of metallurgical transformations in real-time is a natural application of the technique due to its remote sensing character. Ultrasonic waves are generated by a pulsed laser (typically of a few nanoseconds of duration and with energy below one Joule) and the detection is assured by another laser (long pulse or continuous) and an interferometer that, together, probe ultrasonic displacements at the material's surface [1]. This all-optical ultrasonic method allows ultrasonic probing of materials at high temperature at a convenient distance (typically tens of centimeters) on the outside of the furnace. In addition to the non-contact character of the laser-ultrasonic method, the technique is broadband (this is especially interesting for frequency domain analysis and for high resolution in the time domain) and has fewer constraints on material geometry (material surface is the transducer: curved surfaces are allowed).

The present paper discusses the use of the laser ultrasonic technique to real-time monitoring of metallurgical transformations and reviews selected results obtained at IMI/NRC using a laser-ultrasonic system coupled to a Gleeble™ thermo-mechanical simulator.

Description of the laser-ultrasonic system coupled the Gleeble furnace: Resistance heating thermo-mechanical simulators have been widely used on metallurgical research to do physical simulations of industrial thermo-mechanical cycles. But only a few hot-stage-adapted sensors are able to monitor rapidly changing microstructures. Among the slow response techniques are the hot stage electron microscopy (TEM, SEM) and X-Ray and neutron diffraction techniques. Dilatometry is a fast, simple, and widely used technique to sense metallurgical transformations but provides very limited information. Ultrasonic techniques are well known to be highly sensitive to many microstructural parameters like grain size, texture, phase fraction, porosity, inclusions, etc. and can be applied to fast transforming microstructures. Figure 1a illustrates the principle of the laser-ultrasonic technique. The generation laser pulse heats the material's surface and vaporizes a small volume (a few nm of material) that, by a recoil effect, generates an ultrasonic wave that propagates through the material. Simultaneously the detection laser illuminates the sample surface and the reflected (or scattered) light, phase-modulated by the surface motion caused by acoustic disturbances, is demodulated by an interferometer. Figure 1b shows a signal obtained at high temperature with the laser-ultrasonic technique.

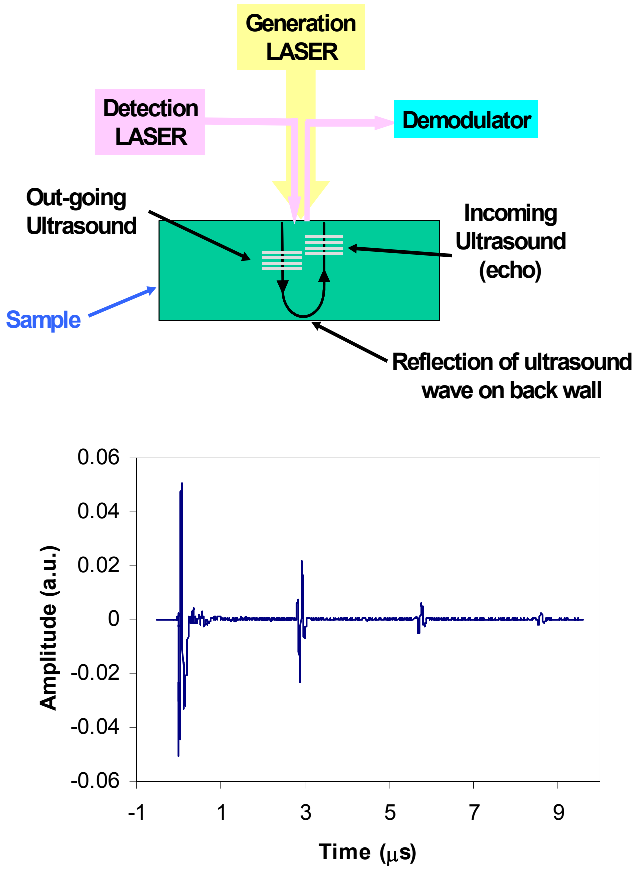

Figure 1. (a) Principle of the laser-ultrasonic technique and (b) a signal obtained in an 8 mm thick steel sample at 1100 °C.

At IMI/CNRC, a laser-ultrasonic system coupled to a Gleeble™ 3500 has been exploited for many years and has demonstrated to be a powerful tool for monitoring microstructure transformations. But that system looks more like a furnace inside a laser-ultrasonic laboratory than a compact sensor coupled to a furnace. More recently considerable effort has been spent to design a cost effective system that could be used by metallurgists as an user-friendly, compact and robust sensor for measurements from outside the furnace. Figure 2 illustrates the concept of such a system where most of elements are in a rack-mount enclosure that is connected to the laser-ultrasonic sensing head by an umbilical cable. The sensing head is to be attached to the furnace and contains a compact generation laser and the focusing optics for both generation and detection lasers. The detection laser and the interferometer are in the rack-mount enclosure and are connected to the sensing head by optical fibers. Also in the rack are the power supplies for the generation and detection lasers, the synchronization electronics and fast signal analog-to-digital converter card and computer. When the head is properly attached to the furnace and when viewing windows have a sufficiently high optical density at the laser wavelengths (such window glasses can easily be purchased to replace existing windows), the system is eye-safe and operators do not need to use laser safety glasses.

Although most of the results presented in this paper were produced before the compact system was available, one could expect similar results since performance tests showed comparable performance to the previous configurations.

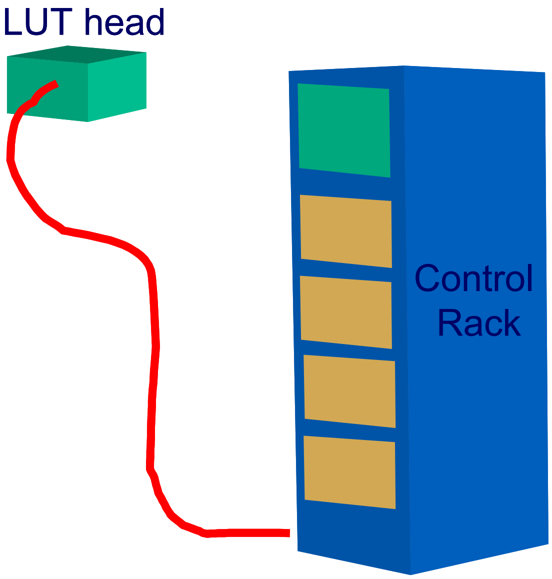

Figure 2 – Concept of a laser ultrasonic system where most components are housed in a rack-mount enclosure. An umbilical cable links the sensing head, which is to be mounted onto the furnace.

Results on Grain Size/Growth: Grain size determination is a classical application of ultrasound to microstructure characterization. The mechanism of acoustic wave scattering by grains is well-known and models for attenuation and velocity in a wide frequency range are available [2]. But often the microstructures are more complex than single phase, log-normal size distribution of equiaxed grains, and neither the definition of grain size nor its relationship to the model grain size parameter is straightforward. Even in this case the ultrasonic evaluation can be very useful, giving a quantitative measurement of how coarse (or fine) is the microstructure. However, laser-ultrasonics coupled to a furnace can monitor grain size evolution *in-situ* and in real time. A very interesting application that has been developed is to monitor grain growth in steel in the high temperature austenitic phase [3-5]. Austenite grain size can determine in a significant extent the mechanical properties of steels, but the determination of the prior austenitic phase grain sizes at room temperature by conventional metallographic methods can be very challenging.

Figure 3 shows the experimental relationship between the attenuation measured at 15 MHz and the austenitic grain size [3]. The grain sizes were determined metallographically after proper cooling from the austenitic phase. In this curve there are various steel grades with different carbon contents. The attenuation was corrected by an experimentally determined temperature factor that includes temperature dependent ultrasonic velocity and absorption as well as possible grain elastic anisotropy dependence on temperature. Considering that the metallographic grain size measurement accuracy is about ±25%, the correlation found in Figure 3 is remarkable. If instead of a single frequency, the attenuation versus frequency curve is used combined with careful image analysis of metallographic images, the spread of the points in the calibration curve is even smaller [6]. The application of the calibration curve of Figure 3 to grain growth monitoring is straightforward and Figure 4 shows an example for a low carbon steel heated at 5 °C/s to 1100 °C and held at this temperature for about 500 seconds while performing the laser-ultrasonic measurements. After proper cooling, the prior austenitic grain size was measured metallographically and was found to be very close to that determined by laser-ultrasound at the end of the thermal cycle as shown in Figure 4. This approach has been also successfully applied to the determination of austenitic grain size in tubes at high temperature in a seamless tube production plant [6].

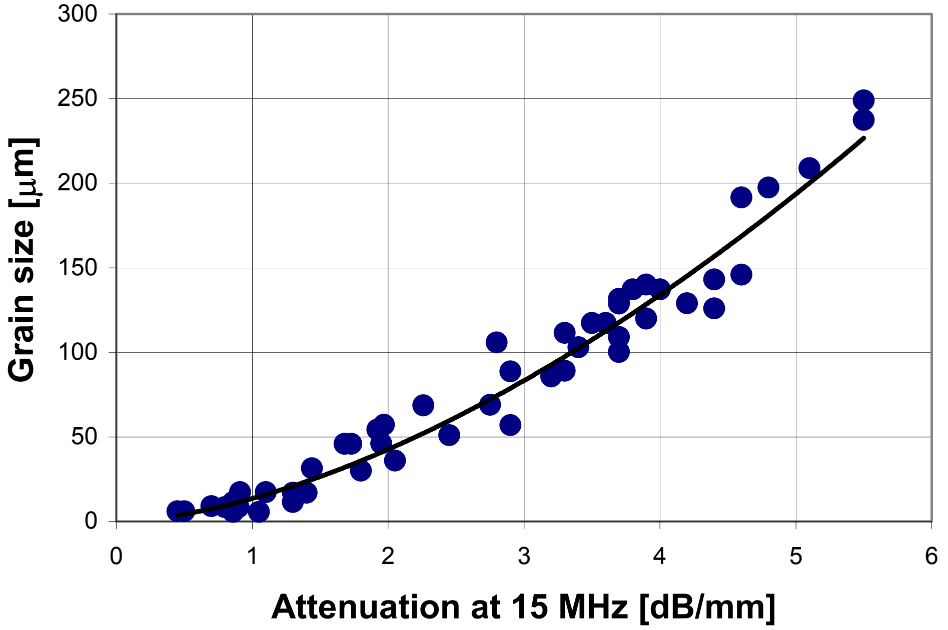

Figure 3. 15 MHz ultrasonic attenuation dependence on average austenitic grain size.

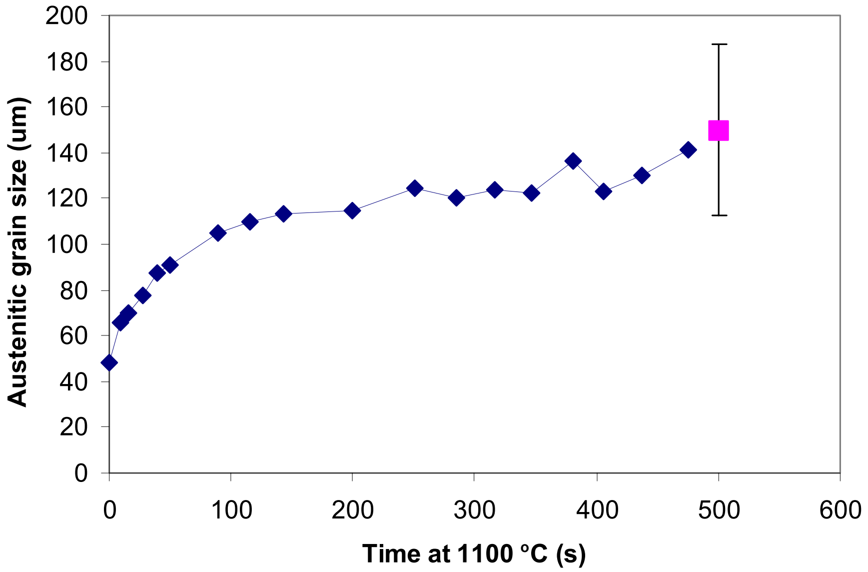

Figure 4. Real-time laser-ultrasonic measurement of austenitic grain sizes at 1100 °C for an A36 steel. The grain size measured by metallography after proper cooling is also shown.

Results on Phase Transformation: Ultrasound can be sensitive to phase fraction also and consequently can be used to monitor phase transformations. While the attenuation can be used to precisely determine the onset of the transformation [7], the velocity has a higher potential to quantitatively evaluate phase fractions during the transformation. The sensitivity of ultrasonic velocity to phase fraction is due to the different elastic constants and/or densities of the different phase components. In addition to classic allotropic phase transformation and second phase precipitation, ultrasound can also be sensitive to magnetic transitions [8,9].

The most classical case of phase transformation is the austenite/ferrite transformation in steels. Figure 5 shows the velocity during austenite decomposition of low carbon steel. The sample was submitted to a thermal cycle that simulates that of heat-affected zone during welding (Rosenthal cycle) with peak temperature of 1350°C and cooling time between temperatures 800 and 500 °C of 30 seconds. The velocity in the austenite phase has an almost linear dependence on temperature and the beginning of the phase transformation at about 560 °C is clearly shown as an abrupt velocity increase. The transformation finishes when the measured velocity attains that of ferrite. The ferrite velocity dependence on the temperature has a more complex (non-linear) dependence on temperature due to the magnetic transition at the Curie temperature (≈ 770 °C) but can be precisely determined during heating of low carbon steels [9]. One limitation of using the ultrasonic velocity to quantitative phase fraction measurements is its sensitivity to texture changes. If during the phase transformation one of the decomposed phases has strong texture, the velocity for this phase could be different from that used for the calibration and accuracy of the evaluated phase fraction is reduced. This limitation can also be reduced by measuring simultaneously the longitudinal and shear wave velocities, which should give important information on texture change. One advantage of using the laser ultrasonic technique instead of the classic dilatometry is the potentiality to measure an absolute phase fraction. This advantage can be particularly useful for retained austenite fraction determination in TRIP or other multiphase steels. Laser-ultrasonics could be also applied to monitoring and quantifying austenite decomposition in a production line.

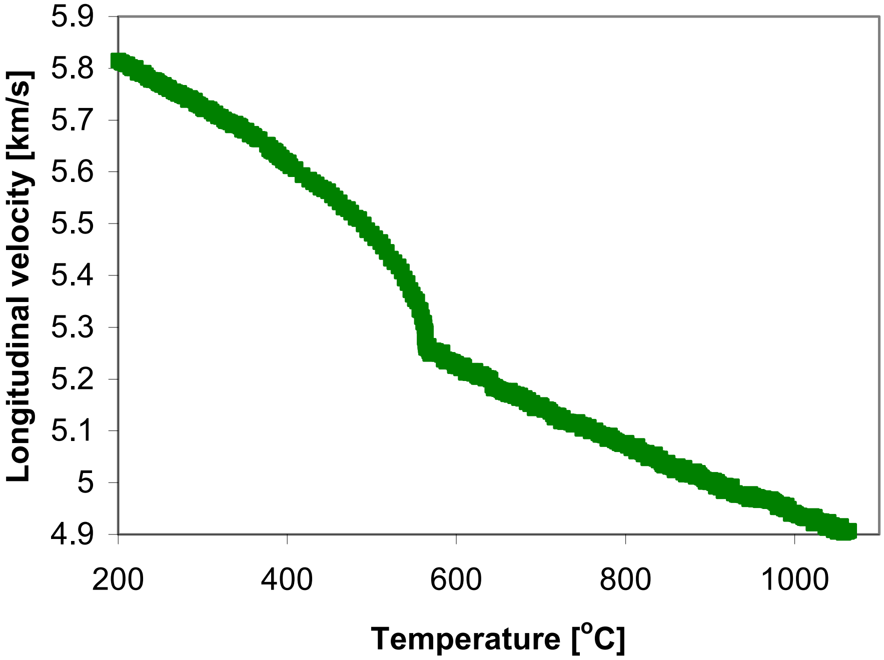

Figure 5. Longitudinal ultrasonic velocity variation during rapid cooling of low carbon steel.

Results on Recrystallization: The recrystallization of cold deformed materials results very often in a texture change and, accordingly, the elastic constants also change. The characterization of texture in sheet of metals can be done in a very convenient way by laser ultrasound resonant spectroscopy [10] and monitoring texture change during the recrystallization could be used to determine in real time the recrystallized fraction. The laser ultrasonic resonant spectroscopy measures simultaneously longitudinal and shear velocities that are directly correlated to the material texture. But often, a single velocity measurement is sufficient to determine the recrystallized fraction.

Figure 6 shows the recrystallized fraction evolution for the aluminum alloy AA5754 cold rolled to a 60% thickness reduction for various annealing temperatures [11,12]. The laser-ultrasonic recrystallization fraction is based on a lever-rule of the longitudinal velocity variation between the cold rolled and the recrystallized states and correlates well with that determined by off-line methods of metallography and yield strength. Similar results have been obtained for other aluminum alloy (AA6111) and recent comparison with neutron diffraction measurements of texture has demonstrated that most of the velocity variation is related to texture changes. During the recrystallization of an interstitial free (IF) steel, the variation of the ultrasonic velocity are very significant as shown in Figure 7. A strong gamma fiber texture is expected to be developed in the final stages of recrystallization and the velocities changes can be directly correlated with this texture component. The velocity change due to the recyrstallization is approximately 5% compared to a typical velocity measurement precision of 0.1%. The magnitude of the velocity variation does not depend on the temperature, as shown in Figure 7, indicating that a simple temperature correction is adequate and the approach can be applied to a continuous heating thermal cycle. For low carbon steels, the texture evolution can be non-monotonic and dependent on annealing temperature [13].

The ultrasonic attenuation is another parameter that can be sensitive to annealing. Although grain size change during recrystallization can change the attenuation, there are evidences that an important part of the attenuation in some materials comes from an absorption mechanism [8,14]. For magnetic materials like steel and nickel, an absorption increase was found during annealing and attributed to a magneto-elastic relaxation mechanism, while for non-magnetic materials like aluminum, a decrease of the absorption can be related to a dislocation motion relaxation mechanism.

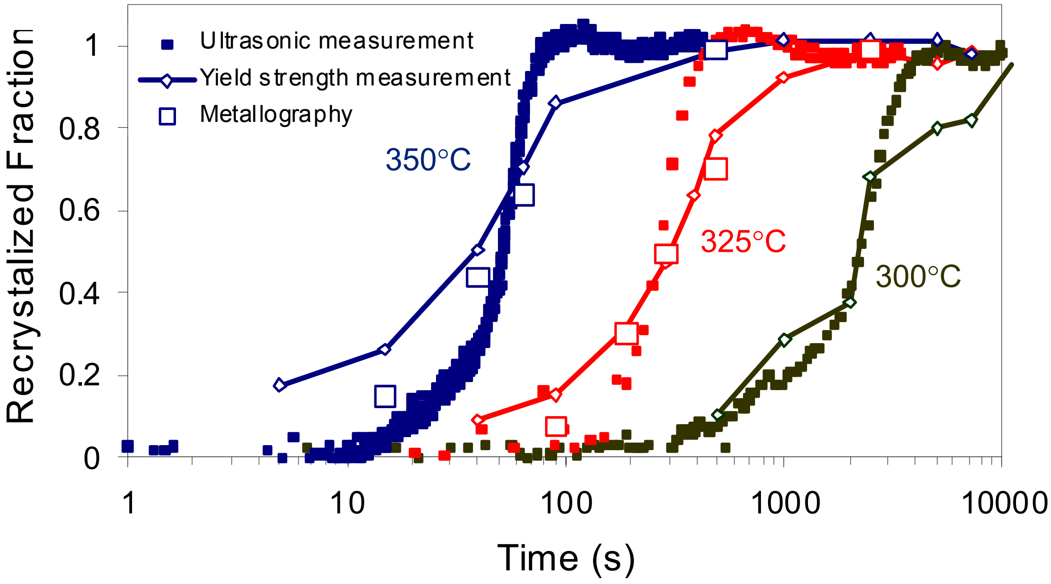

Figure 6. Recrystallized fraction of the aluminum alloy AA5754 determined by laser-ultrasonic velocity, yield strength and metallography.

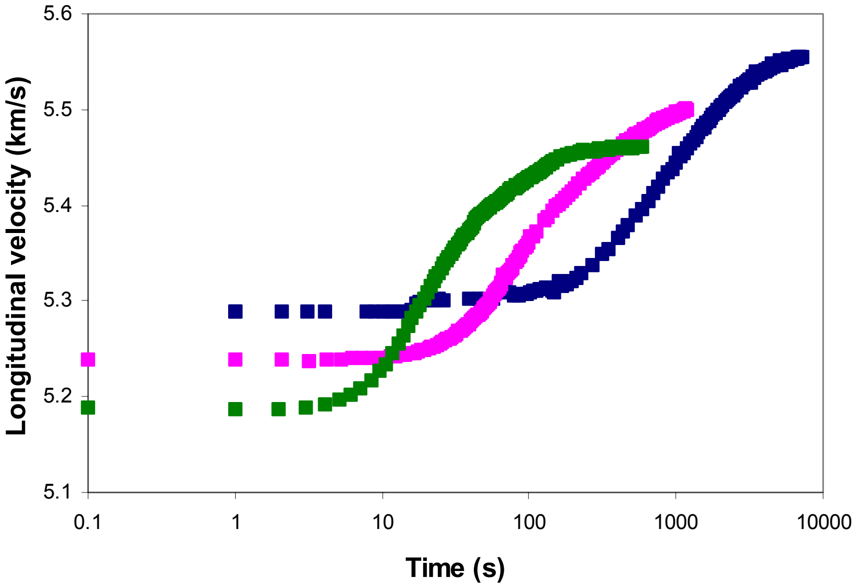

Figure 7. Longitudinal velocity variation during recrystallization of IF steel at various isothermal annealing temperatures.

Discussion: The applications presented (grain growth, phase transformation and recrystallization) are some of the most obvious uses of a laser-ultrasonic system coupled to a thermo-mechanical simulator. Other very useful applications include the precise determination of the elastic moduli [15] of materials or characterization of semi-solid phase transformation [16]. Simultaneous measurements of more than one microstructural parameter are possible when they modify different ultrasonic parameters (eg. attenuation for grain growth or velocity for texture). The most important and most obvious advantage of real-time monitoring of metallurgical transformations by laser-ultrasonics as compared to off-line techniques like metallography and x-ray diffraction is time saving. It provides almost continuous measurements (up to a hundred measurements per second, depending on the laser repetition rate) that eliminate interruptions of the thermal-cycle and subsequent sample preparation for metallographic or other characterization technique. Since the ultrasound propagates through the thickness of the material, the measured property is averaged over the volume where the ultrasound propagates. This can be an interesting advantage over surface measurements. The main limitation of the technique is that few parameters can be measured (velocity, attenuation) and it cannot provide the detailed information provided, for example, by metallography. But for well-calibrated applications, this few parameters character can be an advantage since it facilitates the automation of the measure. Although there are basically only two parameters to be measured, the attenuation and the velocity, additional information can be found in their frequency content. Also two ultrasonic modes can be measured, the longitudinal and transverse, which can probe different characteristics of the material. Other modes like surface waves and plate waves are also possible. Another interesting capability of laser-ultrasonics is the possibility to utilize the technique in production environments and to perform measurements during materials processing [6].

Conclusion: The various results presented on real-time monitoring the metallurgical transformations by laser-ultrasonics demonstrate the great sensibility and flexibility of the technique. These examples of monitoring metallurgical parameters like grain size, phase fraction and recrystallized fraction in materials commonly used in engineering practice are of great practical importance. A compact and cost-effective laser-ultrasonic system coupled to a thermomechanical simulator has been made available and can be a valuable and unique tool for research in metallurgy.

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